

Nitrate-Nitrite, Manual Cadmium Reduction SM 18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> Ed 4500-NO <sub>3</sub> -E					
Relevant Aspect of Standards	Reference	Y	N	N/A	Comments
<i>Records Examined:</i> SOP Number/ Revision/ Date _____ Analyst: _____ Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
Were NO <sub>3</sub> <sup>-</sup> determinations began promptly?	NO <sub>3</sub> <sup>-</sup> A Introduction				
If determinations were not began promptly, were samples stored at 4°C?	NO <sub>3</sub> <sup>-</sup> A Introduction				
Were unchlorinated samples preserved with 2 mL concentrated H <sub>2</sub> SO <sub>4</sub> /L if stored for longer than 2 days?	NO <sub>3</sub> <sup>-</sup> A Introduction				
When samples were preserved with acid, was the determination of NO <sub>2</sub> <sup>-</sup> not needed?	NO <sub>3</sub> <sup>-</sup> A Introduction				
Were turbid samples filtered prior to analysis?	NO <sub>3</sub> <sup>-</sup> A Introduction				
Were samples where copper, iron, and other metal concentrations above several mg/L was suspected treated with EDTA prior to analysis?	4500-NO <sub>3</sub> <sup>-</sup> E 1 b				
Where samples were contaminated by oil and grease, were they solvent extracted prior to analysis?	4500-NO <sub>3</sub> <sup>-</sup> E 1 b				
Were samples checked for residual chlorine, and, if RC was present, were samples treated with sodium thiosulfate?	4500-NO <sub>3</sub> <sup>-</sup> E 1 b				
If a spectrophotometer was used, did it have a wavelength of 543 nm with a path length of 1 cm or longer?	4500-NO <sub>3</sub> <sup>-</sup> E 2 b 1				
If a filter photometer was used, did it have a light path of 1 cm or longer and a transmittance near 540 nm?	4500-NO <sub>3</sub> <sup>-</sup> E 2 b 2				
Did absorbances of reagent blanks never exceed 0.01?	4500-NO <sub>3</sub> <sup>-</sup> E 3 a				
Were 25 g aliquots of 20 to 100 mesh Cd granules washed with 6N HCl?	4500-NO <sub>3</sub> <sup>-</sup> E 3 b				
In repeated cycles were Cd granules next swirled with 2% CuSO <sub>4</sub> solution for 5 minutes or until blue color faded until a brown colloidal precipitate developed?	4500-NO <sub>3</sub> <sup>-</sup> E 3 b				
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Was an EDTA solution prepared by dissolving 13 g NH <sub>3</sub> Cl and 1.7 g disodium ethylenediamine tetraacetate in 1 L water and then adjusting the pH to 8.5?	4500-NO <sub>3</sub> <sup>-</sup> E 3 d				
Was an color reagent prepared by diluting 100 mL 85% phosphate acid and 10 g sulfanilamide and 1 g N-(1-naphthyl)-ethylenediamine to 1 L with reagent water?	4500-NO <sub>3</sub> <sup>-</sup> E 3 c				
Was an ammonium chloride-EDTA solution prepared by diluting 300 mL of the above ammonium chloride-EDTA solution to 500 mL with water?	4500-NO <sub>3</sub> <sup>-</sup> E 3 d				
Was a copper sulfate solution prepared by diluting 20g CuSO <sub>4</sub> •5H <sub>2</sub> O to 1 L with water?	4500-NO <sub>3</sub> <sup>-</sup> E 3 g				
Was a stock nitrate solution prepared by drying KNO <sub>3</sub> at 105°C for 24 hours and diluting 0.7218 g to 1000 mL with H <sub>2</sub> O and then preserving it with 2 mL of CHCl <sub>3</sub> /L?	4500-NO <sub>3</sub> <sup>-</sup> E 3 h				
Was an intermediate nitrate solution prepared by diluting 100 mL of above nitrate solution to 1000 mL with water and preserved with 2 mL of CHCl <sub>3</sub> /L?	4500-NO <sub>3</sub> <sup>-</sup> E 3 i				
Were the nitrate solutions not used for longer than 6 months?	4500-NO <sub>3</sub> <sup>-</sup> E 3 h				
Was a stock nitrite solution prepared by diluting 1.232 g NaNO <sub>2</sub> with water and preserving with 1 mL CHCl <sub>3</sub> and then titrating it to determine its concentration?	4500-NO <sub>3</sub> <sup>-</sup> E 3 j				
Was an intermediate nitrite solution prepared by diluting the volume of above solution necessary to make 50 µg N/ 1.00 mL?	4500-NO <sub>3</sub> <sup>-</sup> E 3 k				
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Was the intermediate nitrite solution prepared daily?	4500-NO <sub>3</sub> <sup>-</sup> E 3 k				
Was a working nitrite solution prepared by diluting 50.0 mL to 500 mL with water?	4500-NO <sub>3</sub> <sup>-</sup> E 3 l				
Did reduction columns first have glass wool plugs inserted into them, then get filled with water, and then have 18.5 cm of Cu-Cd granules added to them?	4500-NO <sub>3</sub> <sup>-</sup> E 4 a				
Were reduction columns prepped by washing with 200 mL of dilute NH <sub>4</sub> Cl-EDTA then about 100 mL of 25% 1.0 mg NO <sub>3</sub> <sup>-</sup> N/L+75% NH <sub>4</sub> Cl-EDTA?	4500-NO <sub>3</sub> <sup>-</sup> E 4 a				
Were sample pHs adjusted to be between 7 and 9 with dilute HCl or NaOH?	4500-NO <sub>3</sub> <sup>-</sup> E 4 b 2				
Were 25 mL sample volumes mixed with 75 mL volumes of NH <sub>4</sub> Cl-EDTA solution?	4500-NO <sub>3</sub> <sup>-</sup> E 4 b 3				
Were the first 25 mL of sample NH <sub>4</sub> Cl-EDTA solution mixtures that passed through the column discarded?	4500-NO <sub>3</sub> <sup>-</sup> E 4 b 3				
If columns were used for more than several hours, were they stored with dilute NH <sub>4</sub> Cl-EDTA solution and not allowed to dry?	4500-NO <sub>3</sub> <sup>-</sup> E 4 b 3				
Was color reagent addition never more than 15 minutes after reduction?	4500-NO <sub>3</sub> <sup>-</sup> E 4 b 4				
Were 2.0 mL of color reagent added to 50 mL volumes of reduced sample mixtures, and the absorbances measured against DI blanks at 543 nm?	4500-NO <sub>3</sub> <sup>-</sup> E 4 b 4				
Were the absorbances of reduced sample mixtures measured between 10 minutes and 2 hours after color reagent addition?	4500-NO <sub>3</sub> <sup>-</sup> E 4 b 4				
Was at least one NO <sub>2</sub> <sup>-</sup> standard compared to a reduced NO <sub>3</sub> <sup>-</sup> standard at the same concentration to verify column efficiency?	4500-NO <sub>3</sub> <sup>-</sup> E 4 c				
Were columns reactivated when above column efficiency dropped below 75%?	4500-NO <sub>3</sub> <sup>-</sup> E 4 c				
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